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Application of Volatile Organic Reference Materials to Ambient Air Monitoring Measurements

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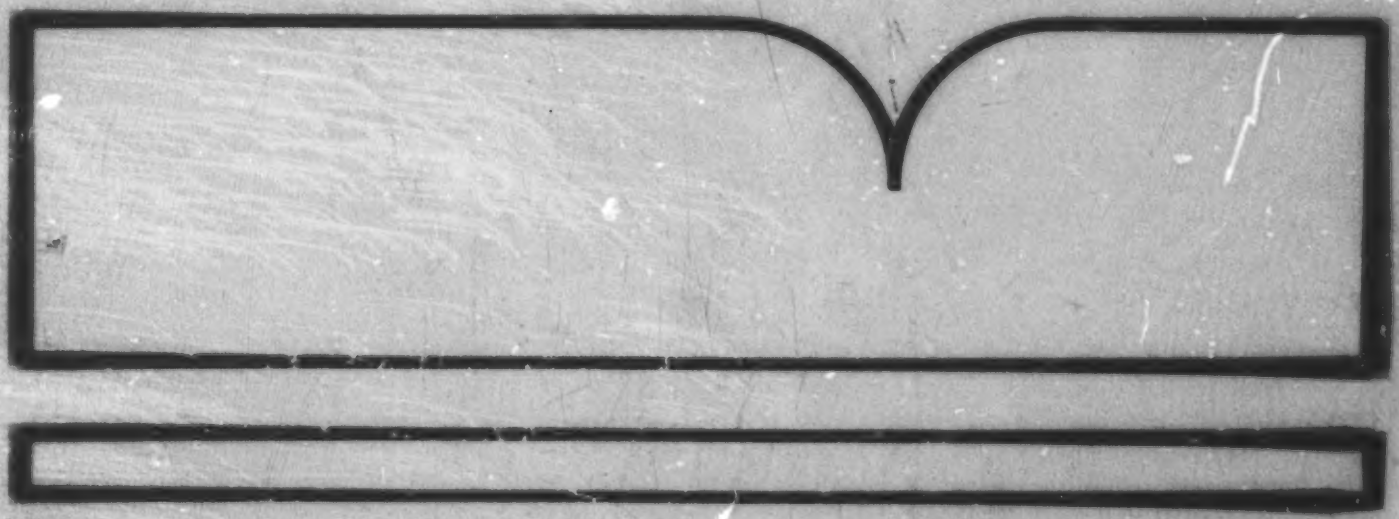

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APPLICATION OF VOLATILE ORGANIC REFERENCE MATERIALS TO AMBIENT AIR MONITORING MEASUREMENTS

by

Howard L. Crist
Environmental Monitoring Systems Laboratory
Research Triangle Park, NC 27711

and

Harry L. Rook
National Bureau of Standards
Washington, DC 20234

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H. L. Crist

ENVIRONMENTAL MONTORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

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16. ABSTRACT

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APPLICATION OF VOLATILE ORGANIC REFERENCE MATERIALS TO AMBIENT AIR MONITORING MEASUREMENTS

Howard L. Crist, U.S. Environmental Protection Agency,
Research Triangle Park, North Carolina 27711

Harry L. Rook, National Bureau of Standards,
Washington, D.C. 20234

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APPLICATION OF VOLATILE ORGANIC REFERENCE MATERIALS TO AMBIENT AIR MONITORING MEASUREMENTS

Introduction

The Environmental Monitoring Systems Laboratory (EMSL) is funding a research program at the National Bureau of Standards (NBS) to develop Standards Reference Materials (SRM's) for certain volatile organic compounds found in ambient air. This effort has resulted in the development of SRM's and other traceable standards in cylinders and permeation tubes. Standards have been prepared at ppm levels for source emission work, and at ppb levels for ambient air measurements. The standards will serve multifunctional needs such as (1) specialized calibration of laboratory instrumentation, (2) validation of experimental data, (3) the preparation of NBS traceable (secondary) standards used for auditing field sampling equipment, and (4) intercomparison of monitoring/analytical methodology.

Standard Reference Materials that have been or will be certified are listed in Table I. The certified values of permeation tubes and cylinder SRM's presently available are valid for one and two years, respectively. Recent laws (Comprehensive Environmental Response, Compensation, and Liability Act and Toxic Substances Control Act) have assigned a high priority to monitoring of toxic organic pollutants in the environment. Of particular concern are the numerous hazardous waste dump sites located in many states. Also the incineration of hazardous wastes is gaining support as an alternative to dumping and is a system that requires close monitoring of organic emissions to assess efficiency. It is likely that the interest created by these developments will result in an increased requirement for standards of organic pollutants. This situation will require the development of additional toxic organic standards with traceability to NBS. It was important to develop a base of primary standards, including preparation and analytical expertise by a rigorous SRM development program for the first volatile organics developed. However, due to the expense ($700-800/cylinder) and time (1-2 years) required to certify and issue SRM's, the direction of future standards development will probably take the form of a more direct approach. The need to provide NBS traceable standards may be met by an RM or CRM standards (see Appendix for definitions) development program.[1] However, a certain amount of flexibility is desirable in the preparation of these standards because of the number of compounds and varying needs for audit standards. Due to the complex and diverse characteristics of hazardous waste sites, gas standards used to audit sampling systems may need to be tailored to fit the particular level and types of substances found. An audit gas mixture can be prepared and analyzed in a short time if the basic technical stability characteristics and behavior of the compounds are known from prior work.

Because of the basic technical investigation performed by NBS in developing volatile organic SRM's, mixtures of gases in cylinders can be prepared with a high degree of accuracy. To further this type of work and provide appropriate audit materials, EPA has established a repository of approximately 40 organic gases at ppm concentrations for use in performance audits of source emissions.[2] This paper discusses the role of traceable gas standards in evaluating ambient air monitoring and analytical systems.

Experimental Section

Apparatus

Samples of high purity liquid organic compounds were weighed by difference in sealed, glass capillary tubes using a Mettler Model ME-22 electrical microbalance. The weights of diluent gas were determined using a Mettler Model PT-15 top-loading balance.

Analyses of the gas mixtures were conducted on a conventional gas chromatograph equipped with a hydrogen flame ionization detector (FID) and an electron capture detector (ECD). The chromatographic separations were accomplished using a 3.7 m x 3.2 mm stainless steel column containing 1% SP-1000 on a 60/80 carbopack B support. The separations were made isothermally at 180°C with a flowrate of 100 cc/min at a head pressure of 55 psig. Samples were injected onto the column using an automatic gas sampling valve with a 10 cc sampling loop made from 6.5 mm copper tubing.

Reagents

All reagents were research-grade purity or better. All organic reagents used as primary standards for gravimetry were also analyzed by GC and by mass spectrometry to verify purity.

Gas Cylinders

The gas cylinders (50 cu. ft.) used in the study were made commercially by Luxfer, Inc. They were constructed of aluminum and were fitted with stainless steel valves with Compressed Gas Association (CGA) 660 outlets. Lead gaskets were used between the mating surfaces of the outlet and the delivery valve.

Gravimetric Gas Mixture Preparation

The high-purity organic reagents used in the preparation of the gas mixture were weighed into a thinwalled, glass, capillary tube approximately 1.6 mm O.D. x 40 mm length. Before the tubes were filled with the organic compound, each end was drawn to a fine point using the flame from a microburner. This was done to minimize both the effect on the weight due to sealing and the chance of trapping significant amounts of liquid in the ends of the tube when adding the contents to an evacuated gas cylinder. One end of the tube was sealed in the flame and the tube was weighed several times against a tare. The empty weight of the tube was established to better than ± 0.5 µg. The tube was then filled by placing the open end of the tube under the surface of a small quantity (≈1-2 mL) of the organic reagent contained in a test tube and partially evacuating the test tube using a tight-fitting syringe. The partial vacuum inside the test tube drew some air out of the capillary tube and, when the vacuum was released, this quantity of air was replaced by the organic liquid. The capillary tube was removed from the test tube, inverted, and centrifuged to force the liquid toward the closed end of the tube. The open end of the capillary tube was then flame-sealed and the tube was reweighed against the same tare. Blank determinations were performed by carrying capillary tubes through the identical procedure except that no organic was used in the test tube.

To transfer the contents of the capillary tube into an evacuated and weighed gas cylinder, the tube was fitted tightly into a short length of Teflon tubing which was then attached to the cylinder. The cylinder valve

was opened slightly and the end of the sample tube nearest the valve was cracked by external force on the Teflon tubing. After most of the organic material had vaporized and been drawn into the cylinder along with the air that leaked past the tube, the other end of the tube was carefully broken in the same way as the front end and atmospheric air was drawn through the remaining portion of the tube to sweep any residual vapor into the cylinder. This process was repeated until all organic components were drawn into the cylinder. The cylinder was then pressurized with high purity nitrogen to 12.4×10^6 Pascals and reweighed.

Results and Discussion

In the spring of 1983, the Quality Assurance Division of EMSL was requested to provide validation for an intercomparison of ambient air monitoring methods for volatile organic pollutants. Three different procedures were compared in the field by having each simultaneously monitor the ambient air in the same urban environment for several days. Each of the methods were different in their approach to sampling and/or analysis. The monitoring systems are described briefly as follows:

(1) Tandem Tubes Using Tenax GC®. Air was simultaneously drawn through four sets of Tenax GC® tubes (13 mm O.D. x 100 mm); each set consisted of two tubes in tandem. Two sets of tubes had the same flow rate (50 ml/min) and the other two sets had a flow rate approximately double that of the other set. Analysis was by GC/MS.

(2) Canister-Cryogenic Trap. Air was collected in a 1 liter canister using a metal-bellows pump until pressurized to 30 psi; an aliquot of the air was then passed through a cryogenic trap to collect organics. The condensate was then volatilized using boiling water into a gas chromatograph equipped with FID and EC detectors.

(3) Single Tube System. Air was simultaneously drawn through two single tubes (6 mm O.D. x 100 mm) containing either Tenax GC® or charcoal; the flow rate of the charcoal tube was approximately four times greater than that of the Tenax tube (50 ml/min). Analysis was by GC/MS.

An adequate performance evaluation of each procedure required an assessment be made of the combined sampling and analytical techniques. To accomplish this objective, NBS prepared and analyzed a mixture of two aromatic and three chlorinated aliphatic compounds in a pressurized cylinder at the concentrations shown in Table II. This gas mixture was then used to deliver known amounts of volatile organic compounds to each ambient air sampling system. The audit cylinder was reanalyzed at intervals of several months to ascertain the stability of the compounds. The results indicated that no significant change in concentration occurred in the gas mixture for as long as a year after preparation. The cylinder contents were analyzed just prior to and after the field audit in order to establish that the amounts delivered to each ambient air monitoring system was verified and did not change during the performance audit. The extremely low concentrations of the audit gas mixture allowed delivery to the collection systems without dilution -- a definite advantage since this avoided a potential source of error. The data in Table II indicate very good consistency of results at these low concentration levels.

Table III contains data obtained by the ambient air monitoring system

that used two Tenax GC® tubes connected in tandem. Significant amounts of benzene, toluene and chloroform were found on the back-up sampling tubes which caused a positive bias when included in the results. An interesting feature of the data is that accuracy for the above compounds improves significantly if the amount reported on the back tube is subtracted from the amount reported on the front tube. This condition can not be attributed solely to adsorbant contamination since analyses of blank sampling tubes did not indicate unusual or excessive amounts of these compounds. Also retention volumes were not exceeded for the front sampling tube so that "breakthrough" to the back tube should not have occurred. A similar phenomenon was observed in the field samples using Tenax GC® tubes in tandem. For reasons that are not entirely understood, this effect is more pronounced for aromatic compounds than for aliphatic chlorocarbons. Consequently, it has been suggested that ambient air sampling using distributed air volumes and single Tenax tubes may be a better approach with fewer uncertainties than a single sampler containing two Tenax tubes in tandem.[3] When the back-up tube is not considered (Table III), the results obtained for the audit gas is in a reasonably narrow range of bias (except for toluene). Thus, the results achieved from the audit indicated support for previously observed phenomena in field data and cast doubts on the usefulness of a tandem sampling tube approach for collection of certain volatile organic compounds.

The results achieved by the pressurized canister-cryogenic trapping technique are given in Table IV. The accuracy was excellent for tetrachloroethylene and reasonably good for chloroform, but results were high for benzene and carbon tetrachloride. Repeatability for all analyses was very good. However, the system failed to detect toluene in the audit gas. This is somewhat perplexing since toluene was detected in the ambient air samples that were analyzed by this technique. This monitoring system is an intermittent sampling approach capable of analyzing several one liter "grab" samples from the air throughout a sampling period.

The laboratory that used single sampling tubes (6 mm O.D. x 100 mm) packed with either Tenax GC® or charcoal reported data that was reasonably accurate only for benzene (Table V). However, at these low concentrations it may not be realistic to expect much better results from monitoring systems using Tenax adsorbant to trap pollutants.

The utility of an NBS traceable audit standard containing volatile organic compounds to conduct performance evaluations of different sampling or analytical methodologies was further demonstrated by the extensive use made of the cylinder over a period of several months. Additional analyses were performed by other analytical laboratories on the contents of the cylinder and the results were generally very good (Table VI). Laboratory A utilized a 10 ml sampling loop to inject the sample into a gas chromatographic column followed by analysis using a flame ionization or electron capture detector. Laboratory B preconcentrated the sample using a cryogenic trap followed by rapid heating to purge the components into a gas chromatograph equipped with a flame ionization or electron capture detector. Laboratory B had a problem with toluene, but otherwise the data shows an expertise in the ability to accurately analyze toxic organic compounds at parts per billion (ppb) concentrations.

Other cylinders were also prepared by NBS that were used to assess the ability of a commercial laboratory to accurately analyze organic volatile pollutants that might be emitted in the vicinity of hazardous waste incinerators. The data indicated that for four of the five components in the cylinder, the laboratory achieved surprisingly accurate results (Table VII).

There was a problem initially with the accurate analysis of vinyl chloride which was subsequently corrected through close consultation between the laboratory and NBS.

Future Reference Materials Development

The future course of reference materials development for assessment of ambient air monitoring procedures will be directed toward increasing the availability of these materials through establishment of a Certified Reference Material (CRM) program. Additionally, the preparation of more highly reactive species in cylinders such as ketones and nitrogen-containing compounds will be attempted. Cylinder standards containing deuterated compounds will be prepared as a means to provide audit materials that can be traced through the sampling system and provide information on the collection efficiencies of solid adsorbants.

Conclusions

It has been shown how NBS organic gas standards can be utilized to establish traceability and assess data from ambient air monitoring sampling and analytical systems. The use of cylinder standards in performance evaluation tests greatly improves the ability to obtain rapid and direct evidence that quality assurance objectives and requirements are being achieved. The uniformity and accuracy of cylinder gas standards allows assessments of performance to be conducted more efficiently. When used as audit materials traceable standards in cylinders assures that all those using the cylinder are compared on an equal basis by removing the uncertainty connected with supplying audit materials that can not be identically prepared (e.g., spiked Tenax GC® cartridges).

Appendix

Definition of Terms

SRM - Standard Reference Material. This is a material that has been characterized by the National Bureau of Standards (NBS) and is issued with a Certificate that gives the results of this characterization. These results are obtained by one of the following certification methods: (1) measurement of the property using a previously validated reference method; (2) measurement by two independent, reliable methods, or (3) measurement of the material by a group of cooperating laboratories, technically competent and thoroughly knowledgeable with the material tested. SRM's are well-characterized, certified materials produced in quantity to improve measurement science. They are prepared and used for three main purposes: (1) to help develop accurate methods of analysis (reference methods); (2) to calibrate measurement systems; and (3) to assure the long-term adequacy and integrity of quality control processes.

RM - Research Material. Unlike SRM's, these are not certified. Instead of a Certificate, RM's are issued with a "Report of Investigation," the sole authority of which is the NBS staff member who authored the report. An RM is intended primarily to further scientific or technical research on that particular material. The principal consideration in issuing an RM is to provide a homogeneous material so that investigators in different laboratories are assured that they are investigating the same material.

CRM - Certified Reference Material. These are reference materials that are issued with a certification of analysis traceable to NBS SRM's. The manufacturer produces the material according to NBS specifications, has

an independent laboratory approved by NBS conduct the necessary analyses using NBS SRM's as calibration standards, and then submits these results to NBS along with the required manufacturing data. NBS then determines if the sample meets the requirements for certification. Generally the manufacturer prepares these materials in quantity and submits several randomly selected samples to the independent laboratory for analysis. This type of material serves to reduce the cost of procuring well-characterized reference materials and at the same time alleviates the shortage of SRM's.

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Table I. AVAILABILITY OF STANDARD REFERENCE MATERIALS FOR VOLATILE ORGANIC COMPOUNDS

Single Component Standards	Year Certified	Container	Nominal Cylinder Conc. ppm or Permeation Rate, µg/min. (25°C)
Benzene	1982	Permeation tube	0.3 - 0.5
	1982	Cylinder	0.25; 10
Tetrachloroethylene	1983	Cylinder	0.25; 9.5
	1984*	Permeation tube[a]	~ 1
Multi-Component Standards			
Benzene Toluene Chlorobenzene Bromobenzene	1984	Cylinde.	Each component at 0.25 and 9.5
Chloroform Tetrachloroethylene Carbon tetrachloride Vinyl chloride	1985	Cylinder	Each component at 0.25 and 9.5

[a]Possible issuance as Research Material

Table II. STABIILTY OF COMPOUNDS IN NBS PREPARED AUDIT CYLINDER, ppb

Compound	Gravimetric Conc., ppb	Analyzed Conc., ppb				Mean of Analyses	Diff.[a] %	Std. Dev.
		4-83	5-83	8-83	4-84			
Benzene	9.0	8.0	8.3	8.6	8.7	8.4	-6.7	0.32
Toluene	6.5	5.9	6.1	6.5	6.5	6.2	-4.6	0.30
Chloroform	3.7	4.6	3.5	4.0	3.8	4.0	8.1	0.46
Tetrachloroethylene	12.4	13.8	11.4	12.6	12.4	12.6	1.6	0.98
Carbon tetrachloride	8.2	8.0	7.3	8.5	8.2	8.0	-2.4	0.51

[a]Difference between the gravimetric concentrations and the mean of analyses.

Table III. SELECTED RESULTS FROM DISTRIBUTED AIR VOLUME MONITORING SYSTEM (TANDEM TENAX TUBES)

Compound	Amount Delivered, ng	Amount Reported, ng		Accuracy, %	
		Front	Back	Front	Total
Benzene	43.4	55.8	22.5	28.6	80.4
Toluene	37.6	58.9	58.0	56.6	211
Chloroform	28.9	35.4	18.0	22.5	84.8
Tetrachloroethylene	131	151	7.5	15.3	21.0
Carbon tetrachloride	75.6	57.1	N.D.	-24.5	-24.5
Benzene	87.5	110	23.8	25.7	52.9
Toluene	75.9	124	44.6	63.4	122
Chloroform	56.9	63.4	12.9	11.4	34.1
Tetrachloroethylene	265	308	5.5	16.2	18.3
Carbon tetrachloride	152	114	N.D.	-25.0	-25.0
Benzene	166	185	21.4	11.4	24.3
Toluene	144	185	36.8	28.5	54.0
Chloroform	108	108	15.5	0.0	14.4
Tetrachloroethylene	503	577	4.4	14.7	15.6
Carbon tetrachloride	290	194	22.2	-33.1	-25.4

N.D. = Not Detected

Table IV. DATA FROM PRESSURIZED CANISTER - CRYOGENIC TRAPPING SYSTEM (GC/FID AND GC/EC ANALYSIS)

Compound	Audit Gas Conc., ppb	Conc. Reported, ppb			Accuracy, %		
Benzene	9.0	17.3	16.8	16.9	98.9	93.1	94.3
Toluene	6.5	NR	NR	NR	---	---	---
Chloroform	3.7	4.4	4.5	4.5	15.8	18.4	18.4
Tetrachloroethylene	12.4	12.2	12.4	12.5	-1.6	0.0	0.8
Carbon tetrachloride	8.2	11.8	11.6	11.6	53.2	50.6	50.6

NR = not reported

Table V. RESULTS FROM AMBIENT AIR MONITORING SYSTEM USING SINGLE SAMPLING TUBES

Compound	Gravimetric Conc., ppb	Reported Conc., ppb		Accuracy, %	
		Tenax	Charcoal	Tenax	Charcoal
Benzene	9.0	7	6	-22.2	-33.3
Toluene	6.5	(a)	(a)	---	---
Chloroform	3.7	2	<1	-45.9	---
Tetrachloroethylene	12.4	6	6	-51.6	-51.6
Carbon tetrachloride	8.2	2	<1	-75.6	---

[a]Not reported because of high backgound on blanks

Table VI. ADDITIONAL ANALYSES OF NBS AUDIT GAS MIXTURE

Compound	Gravimetric Conc., ppb	Analyses, ppb		Accuracy, %	
		Lab A	Lab B	Lab A	Lab B
Benzene	9.0	9.0	10.7	0	18.9
Toluene	6.5	(a)	10.7	---	64.6
Chloroform	3.7	3.0	3.8	-18.9	2.7
Tetrachloroethylene	12.4	11.8	13.8	-4.8	11.3
Carbon tetrachloride	8.2	7.4	8.1	-9.8	-1.2

[a]Toluene was not analyzed because it was not a target compound in the study for which the audit was performed.

Table VII. EVALUATION OF CAPABILITIES FOR ANALYSIS OF ORGANICS NEAR HAZARDOUS WASTE INCINERATORS USING NBS TRACEABLE AUDIT MIXTURES

Compound	Concentration, ppb		Accuracy, %
	Gravimetric	Contractor Analysis	
Chloroform	23	24	4.3
	10	10	0.0
	136	131	-3.7
	124	117	-5.6
Carbon tetrachloride	20	22	10.0
	19	20	5.3
	154	164	6.5
	140	148	5.7
Tetrachloroethylene	28	30	7.1
	23	23	0.0
	145	149	2.8
	126	128	1.6
Benzene	18	19	5.6
	17	18	5.9
	192	191	-0.5
	132	136	3.0
Vinyl chloride	27	35	29.6
	16	30	87.5
	192	212	10.4
	151	161	6.6